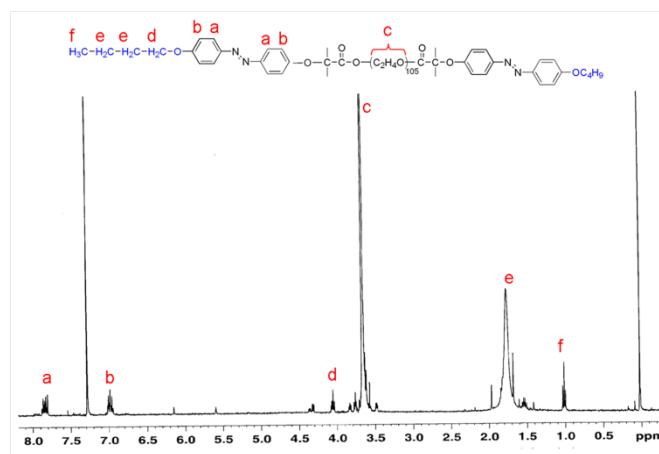


## Supporting Information

### Photo-driven Pulsating Vesicles

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**Fig. S1**  $^1\text{H}$ -NMR spectrum of  $(\text{C}_4\text{H}_9\text{O}-\text{Azo})_2\text{-PEG}$  in  $\text{CDCl}_3$

**Table S1** DLS results of the self-assembly behavior for  $(\text{C}_4\text{H}_9\text{O}-\text{Azo})_2\text{-PEG}$  in THF/water mixed solvent with different volume ratios (Values are mean  $\pm$  SD)

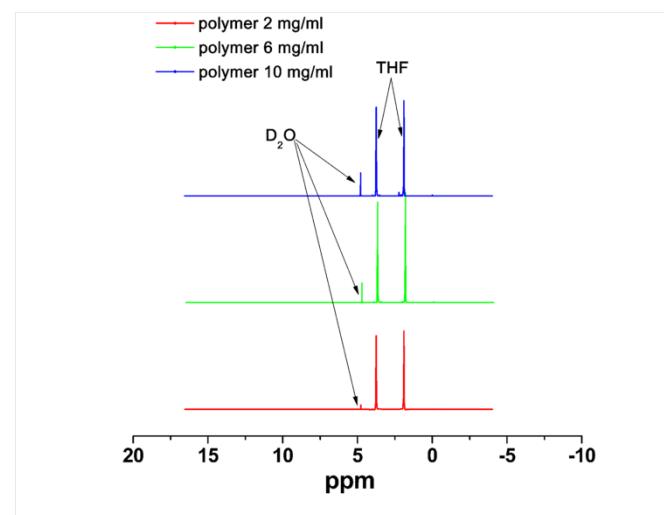
THF / water	1:10	1:9	1:8	1:7	1:6	1:5	1:4	1:3	1:2	1:1
DLS test done on the 1 <sup>st</sup> day when the vesicles are prepared										
$D_h$ (nm)	239 $\pm$ 15	273 $\pm$ 16	262 $\pm$ 50	405 $\pm$ 22	432 $\pm$ 32	545 $\pm$ 29	501 $\pm$ 9	538 $\pm$ 154	618 $\pm$ 112	1003 $\pm$ 68
PDI	0.13 $\pm$ 0.1	0.15 $\pm$ 0.02	0.16 $\pm$ 0.1	0.13 $\pm$ 0.05	0.11 $\pm$ 0.05	0.07 $\pm$ 0.05	0.05 $\pm$ 0.01	0.1 $\pm$ 0.1	0.14 $\pm$ 0.1	0.3 $\pm$ 0.1
DLS test done 30 days after the vesicles are prepared										
$D_h$ (nm)	201 $\pm$ 28	248 $\pm$ 26	217 $\pm$ 25	608 $\pm$ 32	502 $\pm$ 27	580 $\pm$ 25	524 $\pm$ 8	675 $\pm$ 52	754 $\pm$ 42	1415 $\pm$ 210
PDI	0.22 $\pm$ 0.1	0.21 $\pm$ 0.1	0.24 $\pm$ 0.05	0.25 $\pm$ 0.1	0.26 $\pm$ 0.1	0.19 $\pm$ 0.05	0.14 $\pm$ 0.05	0.15 $\pm$ 0.1	0.27 $\pm$ 0.1	0.33 $\pm$ 0.2

**Table S2** Summary of Laser Light Scattering Results<sup>a</sup>

Sample	R <sub>h</sub> (nm)	R <sub>g</sub> (nm)	R <sub>g</sub> /R <sub>h</sub>	M <sub>w</sub> g/mol (aggregates)	M <sub>w</sub> g/mol (polymer)	N <sub>aggregation</sub> <sup>b</sup>
(H-Azo) <sub>2</sub> -PEG	252	299	1.19	7.43×10 <sup>7</sup>	7.69×10 <sup>3</sup>	1.03×10 <sup>4</sup>
(CN-Azo) <sub>2</sub> -PEG	273	334	1.22	7.95×10 <sup>7</sup>	7.75×10 <sup>3</sup>	1.03×10 <sup>4</sup>
(C <sub>4</sub> H <sub>9</sub> O-Azo) <sub>2</sub> -PEG	250	318	1.27	8.20×10 <sup>7</sup>	7.93×10 <sup>3</sup>	1.05×10 <sup>4</sup>

<sup>a</sup> Test on dark-adapted samples; <sup>b</sup>N<sub>aggregation</sub>=M<sub>w</sub>(aggregates)/M<sub>w</sub>(polymer), where the values of

M<sub>w</sub>(polymer) are from GPC analysis.



**Fig. S2** <sup>1</sup>H NMR spectra changes of (C<sub>4</sub>H<sub>9</sub>O-Azo)<sub>2</sub>-PEG polymer in THF/D<sub>2</sub>O (vol 1:4) with different polymer concentrations. The signal at δ 4.8 ppm is assigned to D<sub>2</sub>O; the signal at δ 3.7 ppm and 1.8 ppm are assigned to THF.

**Table S3** Summary of  $^1\text{H}$  NMR Results of **Fig. S2**

Polymer Concentration	$I_1 : I_2 : I_3^*$	$I_2 / I_1$ (or $I_2 / I_3$ )
2 mg/ml	0.01 : 1 : 1	100
6 mg/ml	0.03 : 1 : 1	33
10 mg/ml	0.06 : 1 : 1	17

\* The  $I_1$  (at  $\delta$ 4.8 ppm,  $\text{D}_2\text{O}$ ),  $I_2$  (at  $\delta$ 3.7 ppm, THF) and  $I_3$  (at  $\delta$ 1.8 ppm, THF), are the integral peak intensities of  $\text{D}_2\text{O}$  and THF.